

II. Rejection Under 35 U.S.C. § 102(e)

Claims 16-30 have been rejected under 35 U.S.C. § 102(e) as anticipated by *Sonoda et al.* (U.S. Patent No. 5,707,732) in view of *Betso et al.* (U.S. Patent No. 6,262,161), which is used to "show the state of the art." See page 2 of the present Office Action.¹ Applicants traverse this rejection for at least the reasons of record and the following additional reasons.

A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference. See M.P.E.P. § 2131, quoting Verdegaal Bros. v. Union Oil Co. of California, 2 U.S.P.Q.2d 1051, 1053 (Fed. Cir. 1987).

In the present case, *Sonoda et al.* fails to disclose each and every limitation of Applicants' claimed invention, either expressly or inherently. For example, *Sonoda et al.* fails to disclose at least (1) natural magnesium hydroxide and (2) hydrolyzable organic silane groups grafted onto the polymer chain for compatibilization of the natural magnesium hydroxide with the polymeric components, as recited by Applicants' claims. Accordingly, Applicants maintain that *Sonoda et al.* fails to anticipate the present claims.

(1) Natural magnesium hydroxide.

Applicants maintain that *Sonoda et al.* does not anticipate claims 16-30 because it fails to teach the inclusion of natural magnesium hydroxide. Rather, *Sonoda et al.* teaches that "[w]hile conventional off-the-shelf magnesium hydroxide and alumina

¹ Applicants acknowledge the withdrawal of claims 16-30 over the same references under 35 U.S.C. § 102(b). See page 2 of the present Office Action.

trihydrate **can** be used, a **preferred** magnesium hydroxide has. . . [specific] characteristics " See col. 6, lines 61 - 64 (emphasis added). These recited characteristics, which are relevant only to crystalline magnesium hydroxide, dictate that the preferred magnesium hydroxide is synthetic and not natural. See specification at page 13, lines 19-26. *Sonoda et al.*'s "preferred magnesium hydroxide and a method for its preparation are disclosed in U.S. Pat. No. 4,098,762." See col. 7, lines 1-3. As discussed in the Response filed January 24, 2003, this again establishes that the preferred magnesium hydroxide is **synthetic** magnesium hydroxide.

Moreover, *Sonoda et al.*'s vague disclosure of "conventional off-the-shelf magnesium hydroxide" fails to anticipate the presently claimed natural magnesium hydroxide. A species is anticipated if one of ordinary skill in the art is able to "at once envisage" the specific species within the disclosed genus. See e.g., M.P.E.P. § 2131.02. One may look to the preferred embodiments to determine which compounds can be anticipated. *Id.*, citing In re Petering, 301 F.2d 676, 133 U.S.P.Q. 275 (CCPA 1962).

In the present case, the term "natural magnesium hydroxide" according to the present invention refers to "magnesium hydroxide obtained by grinding minerals based on magnesium hydroxide, such as brucite and the like." See specification at page 11, line 34 - page 12, line 2. One of ordinary skill in the art would not be able to "at once envisage" such magnesium hydroxide from *Sonoda et al.*'s mere recitation of a genus as undefined as "conventional off-the-shelf magnesium hydroxide." See M.P.E.P. § 2131.02 (citing Akzo N.V. v. International Trade Comm'n, 808 F.2d 1471, 1 U.S.P.Q.2d 1241 (Fed. Cir. 1986) (Claims to a process for making aramid fibers using a 98%

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solution of sulfuric acid were not anticipated by a reference which disclosed using sulfuric acid solution but which did not disclose using a 98% concentrated sulfuric acid solution)). Moreover, *Sonoda et al.*'s preferred embodiments teach the use of synthetic magnesium hydroxide. Accordingly, for at least these reasons, Applicants maintain that *Sonoda et al.* fails to anticipate the present claims.

Indeed, even the Office is not willing to assert that *Sonoda et al.* teaches natural magnesium hydroxide. See pages 2-4 of the Office Action. Rather, in the present Office Action, the Examiner has asserted that "*Sonoda* uses his magnesium [hydroxide] to obtain the same properties and purpose as does applicants, namely, to impart flame-retardant properties." See page 4 of the present Office Action. However, whether or not natural magnesium hydroxide and synthetic magnesium hydroxide are used to obtain the same properties and purpose is legally irrelevant in a rejection under 35 U.S.C. § 102. As discussed above, *Sonoda et al.* fails to teach the presently claimed natural magnesium hydroxide, either expressly or inherently. No inquiry beyond this determination is appropriate because the Examiner has elected to make a Section 102 rejection and not a Section 103 rejection.

Nevertheless, Applicants would like the Examiner to be aware that her assertion is factually erroneous. While both natural magnesium hydroxide and synthetic magnesium hydroxide can be used as flame-retardant fillers, and thus may be ultimately used for the same intended purpose, each can provide different advantages and disadvantages to compositions in which they are comprised. For example, as discussed in Applicants' specification, natural magnesium hydroxide has a low affinity with polymer material, in particular when the polymer is of low polarity, as in the case of

polyolefins. See page 2, lines 25-30. Moreover, as discussed in Applicants' specification, EP-780,425 points out that the presence of different metal impurities in magnesium hydroxide of natural origin may cause degradation of the polymer matrix into which the magnesium hydroxide is dispersed. See page 2, lines 31-35, page 12, lines 22-28. "Therefore, research efforts have been directed towards **modifying properties** of magnesium hydroxide to improve its compatibility with the polymer matrix and its degree of purity. Various **synthetic methods** have thus been developed...." See specification at page 3, lines 1-5 (emphasis added). Accordingly, Applicants submit that natural magnesium hydroxide and synthetic magnesium hydroxide can provide different advantages and disadvantages to compositions in which they are comprised.

Similarly, the Examiner's assertion that "there is no clear factual evidence on this record of unexpected or superior properties...when natural magnesium hydroxide [is used] instead of synthetic magnesium hydroxide" is also inappropriate in a rejection under 35 U.S.C. § 102. "Evidence of secondary consideration, such as unexpected results or commercial success, is **irrelevant to 35 U.S.C. 102 rejections** and thus cannot overcome a rejection so based." See M.P.E.P. § 2131.04 (emphasis added). Nevertheless, Applicants submit that the specification unequivocally establishes that the problems of using natural magnesium hydroxide were unexpectedly overcome in the compositions, as claimed, and unexpectedly yielded improved mechanical properties. See, e.g., specification at page 6, lines 7-26.

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Since *Sonoda et al.* does not teach the presently claimed natural magnesium hydroxide, Applicants submit that the Section 102 rejection is improper and should be withdrawn.

(2) Hydrolyzable organic silane groups grafted onto the polymer chain for compatibilization of the natural magnesium hydroxide with the polymeric components.

Applicants maintain that *Sonoda et al.* does not expressly or inherently disclose hydrolyzable organic silane groups grafted onto the polymer chain for compatibilization of the natural magnesium hydroxide with the polymeric components, as presently claimed. First, as discussed above, *Sonoda et al.* fails to teach natural magnesium hydroxide and therefore it does not and cannot teach hydrolyzable organic silane groups for compatibilization of natural magnesium hydroxide with polymeric components as presently claimed.

Second, as detailed in Applicants' January 24, 2003, Response and again below, even assuming, *arguendo*, that *Sonoda et al.* discloses the presently claimed natural magnesium hydroxide, *Sonoda et al.*'s disclosure of optionally grafting a copolymer with an alkenyl trialkoxy silane in the presence of organic peroxide **IS NOT** a disclosure of a "hydrolyzable organic silane groups grafted onto the polymer chain for compatibilization of the natural magnesium hydroxide with the polymeric components," as presently claimed. See col. 6, lines 41-44. *Sonoda et al.*'s silane compounds are not present for compatibilization but rather are present for a wholly separate and different purpose. Hence, there **CANNOT** be anticipation.

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The Examiner has asserted that the recited limitation "is not more than the requirement that the organic silane component function in its normal capacity, i.e.,...function as a coupling agent." See page 3 of the present Office Action. The Examiner goes on to say, in reliance on *Betso et al.*, that "[t]he coupling agent can be grafted onto the interpolymer and serves to couple the filler to the interpolymer. Thus this property is an inherent function of the organic silane component." *Id.* These assertions evidence a failure to appreciate the full and complete teachings of *Sonoda et al.*

Here, the Examiner's inherency argument relies upon *Sonoda et al.*'s teaching of a silane grafted to the polymer and *Betso et al.*'s teaching that there may be a coupling between the interpolymer and the magnesium hydroxide. This argument fails because of two faulty presumptions: (1) that *Betso et al.*'s teachings are adequate to establish inherency and (2) that *Sonoda et al.*'s silane grafts act as a compatibilizer.

With regard to the first presumption, the Federal Circuit has explained that "[i]nherency may **not be established by probabilities or possibilities**. The mere fact that a certain thing may result from a given set of circumstances is **not** sufficient." Continental Can Co. U.S.A., Inc. v. Monsanto Co., 20 U.S.P.Q.2d 1746, 1749 (Fed. Cir. 1991) (emphasis added). The result or characteristic must **necessarily** be present in the prior art to establish the inherency of that result or characteristic. See M.P.E.P. § 2112 (*citing In re Rijckaert*, 28 U.S.P.Q.2d 1955, 1957 (Fed. Cir. 1993)). Thus, the question is whether *Betso et al.* teaches what must necessarily occur (inherency) or what may possibly occur (not inherency).

A reading of *Betso et al.* clearly establishes the latter rather than the former.

Betso et al. states:

[w]hile not wishing to be bound to any **theory**, it is **believed** that **under some conditions** component (C)(3) [the at least one coupling agent] **may** become coupled, i.e., grafted, to the interpolymer or another polymer of the composition capable of such grafting. Similarly, component (C)(3) **may** become coupled, i.e., bound to at least one filler. Thus, component (C)(3) often serves as a coupling agent which **may** couple the following components:

(a) **interpolymers or polymers to other interpolymers or polymers;**

(b) **fillers to other fillers;**

(c) fillers to interpolymer or polymers; **or**

(d) combinations thereof. It is **believed** that the interpolymer is typically grafted onto the coupling agent via a vinyl group while an active polar group on the coupling agent usually binds the coupling agent to the filler.

See col. 10, lines 42-58 (emphasis added). Hence, *Betso et al.* merely provides several hypotheses regarding the function of the coupling agent. According to *Betso et al.*, the coupling agent may just as likely couple the polymers together. By definition, this is not inherency. See M.P.E.P. § 2112.

Moreover, *Betso et al.*'s statement that "[t]he extent of coupling, if any, varies depending on the amount and types of ingredients and the conditions to which the ingredients are subjected" (see col. 10, lines 59-61) further establishes the lack of inherency. In fact, *Betso et al.* teaches that under certain circumstances there will be no compatibilization. See col. 10, lines 59-61. For example, moisture curing is a well-known technique to crosslink polymers using water to hydrolyze silyl groups on a polymer backbone, which then undergo condensation reactions to form crosslinks via Si-O-Si bond formation. Accordingly, conditions under which condensation of the trialkoxysilyl groups occurs and timing of the same will have an effect on the coupling, if

any. Similarly, the order of the steps of curing the polymer and of combining the grafted polymer with magnesium hydroxide will affect coupling. As the trialkoxysilyl groups mentioned in *Sonoda et al.* are intended to serve as crosslinking sites between copolymers, rather than between a copolymer and a filler, it is not completely clear that the process and order of steps described in *Sonoda et al.* for making the compositions would necessarily result in coupling with magnesium hydroxide. Nothing in *Sonoda et al.* suggests a process whereby compatibilization, rather than cross-linking, inherently occurs.

It is a simple matter of fact that the mere presence of a component does not mean it will inherently function as Applicants claim. See Ex parte Schricker, 56 U.S.P.Q.2d 1723, 1725 (Bd. Pat. App. & Int. 2000) (Board vacating an obviousness rejection, which had merely stated the claimed effect would be inherent from the use of a specific compound). Thus, *Betso et al.* does not teach Applicants' limitation to be inherent to the presence of grafted silane groups.

With regard to the second presumption, Applicants direct the Examiner to *Sonoda et al.*'s teachings regarding: (1) *Sonoda et al.*'s purpose for the trialkoxysilyl groups on the polymer backbone and (2) *Sonoda et al.*'s requirement of anhydride-derived carboxyl groups on the polymer backbone.

Sonoda et al. discloses that the each of its three copolymer types can be made **hydrolyzable** by grafting the copolymer with, for example, an alkenyl trialkoxy silane in the presence of an organic peroxide and then **moisture cured** (i.e., cross-linked) in the presence of a silanol condensation catalyst. See col. 6, lines 41-59. In fact, moisture curing is a well-known technique to **crosslink** polymers. See for example, A Review of

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Fifteen Years Development in Moisture Curable Copolymers and a Future Outlook,

http://www.borealisgroup.com/public/pdf/customer_centre/WC_Mumbai2002_Visico.pdf
(courtesy copy submitted herewith), which discloses that *Sonoda et al.*'s process at col. 6, lines 28-56 is well-known to create crosslinking in copolymers.

Notably, this use for silane grafts contradicts the use of silane grafts in Applicants' claims, wherein, "[b]y suitably dosing the amount of the silane groups, this performance [i.e., compatibilization] is achieved **without causing an appreciable cross-linking of the polymer matrix.**" See specification at page 6, lines 21-23. Because *Sonoda et al.*'s trialkoxysilyl groups are only intended to crosslink polymers and not to couple them with magnesium hydroxide, for this reason alone there is no express or inherent disclosure of the compatibilization limitation.

Moreover, as discussed in the Response filed January 24, 2003, *Sonoda et al.* **requires** that at least one of its polymers (i) and (ii) be modified with an anhydride of an unsaturated aliphatic diacid. See e.g., col. 2, lines 1-3. Such "maleic anhydride resins can be used as coupling agents between polymers -- mainly polyolefins such as polyethylene and polypropylene -- and fillers, in order to increase the filler acceptability of polymers." See Dupont Industrial Polymers: Fusabond®, <http://www.dupont.com/industrial-polymers/fusabond/H-81624/H-81624.html> (courtesy copy previously submitted). The fact that the anhydride is present as a coupling agent is further supported by the data in Table 2 of the specification, which reports improved properties from the use of silane over the use of an anhydride as a coupling agent.

In addition, Applicants' specification teaches that, as "compared to unsaturated aliphatic diacid anhydrides, organic silanes...[are] less reactive." See specification at

page 6, lines 27-30. Accordingly, Applicants submit that one of ordinary skill in the art would recognize that in compositions where both compounds are present, such as *Sonoda et al.*, the anhydrides would compatibilize with the magnesium hydroxide and the polymer, and the organic silanes would be available to crosslink the polymers in the presence of a catalyst, as dictated by *Sonoda et al.* (see col. 6, lines 28-56). Thus, again, there is no express or inherent disclosure of compatibilization.

For at least the foregoing reasons, Applicants maintain that *Sonoda et al.* fails to disclose each and every limitation of Applicants' claimed invention, either expressly or inherently, and therefore that *Sonoda et al.* fails to anticipate the present claims.

III. Conclusion

In view of the foregoing remarks, Applicants respectfully request the Examiner's reconsideration and reexamination of the application, and the timely allowance of the pending claims.

Please grant any extensions of time required to enter this response and charge any additional required fees to Deposit Account No. 06-0916.

Respectfully submitted,

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A REVIEW OF FIFTEEN YEARS DEVELOPMENT IN MOISTURE CURABLE COPOLYMERS AND A FUTURE OUTLOOK

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1. INTRODUCTION

Crosslinkable ethylene-vinyl silane copolymers (EVS) specific for the global wire and cable market, were introduced fifteen years ago. It is the intention of this paper to review the compound and its application development, which has been taking place in the intervening years. This development has given moisture curable copolymers a dominant position as insulation materials for 90°C rated 1kV cables.

Application fields for these types of material are broadening and competitive technologies have more recently been developed for medium voltage, track resistant, insulated overhead and flame retardant cables.

1.1 Moisture curing processes

Competitive techniques for crosslinking of low voltage cables arrived in 1968 with the introduction of Sioplas™, the first moisture crosslinkable grafting process⁽¹⁾, see figure 1. This was followed a few years later by Monosil™, the one step grafting process⁽²⁾. In these processes vinyl silane, normally vinyl-trimethoxysilane (VTMS), is grafted onto a polyethylene molecule.

process originally developed for the production of low density polyethylene (LDPE). In all moisture curable processes a crosslinking agent, originally an organo-tin derivative such as di-n-butyltin-dilaurate (DBTDL), has to be added. In the copolymer and Sioplas processes it is added via a catalyst masterbatch and in the Monosil process it is pumped into the extruder. In all these processes the crosslinking was originally performed "off line" by introducing the cable core drum into a steam cabinet or a water bath heated to 70-90°C for 6-48 hours for 1 kV cables.

The copolymer process solved some of the problems associated with the older silane technologies. As it improved the product consistency and resulted in a cable manufacturing without any handling of explosive and burnable liquids. Copolymers have a superior storage stability and contains no volatiles. It also resulted in reduced investments as there is no need for specially designed extruders/compounding units as is the case for processes require the chemical grafting operation to take place.

Accordingly the copolymer process made it possible to use ordinary polyethylene and PVC extrusion lines, without any modification. The only requirement is the addition of a small feeder feeder for adding catalyst masterbatch (normally 5-10%) and preferably a dryer for masterbatches.

Some unexpected weaknesses of moisture curable copolymers were, however, also soon discovered. The first generation of moisture curable compounds had difficulty matching the extrusion characteristics and curing speed of the grafted materials.

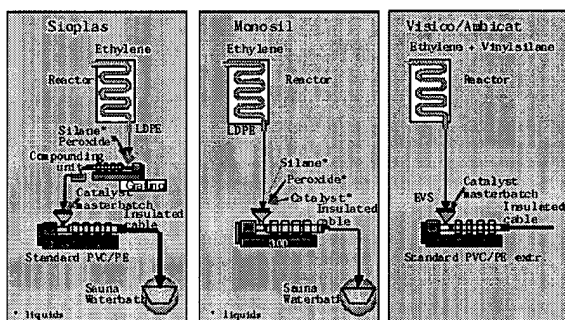


Figure 1. Silane crosslinking processes

In the silane copolymer process, vinyl silane units necessary for the crosslinking are copolymerised with ethylene using the traditional high-pressure

FUNDAMENTAL DEVELOPMENTS OF THE COPOLYMER TECHNOLOGY

1.1 Polymerisation

EVS copolymers are produced by free radical polymerisation in accordance with the original polyethylene polymerisation process. The so called "high pressure process", invented already 1933. Together with ethylene some minor amount of a vinyl silane, as well as some trace amounts of a radical initiator e.g. oxygen is fed into the reactor. The generated radicals start a polymerisation at a pressure of 2-3000 bar and temperatures of 200-350°C.

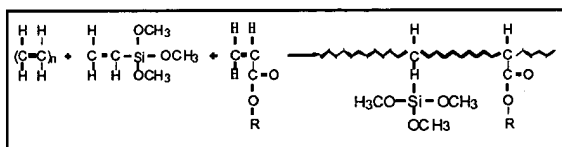


Figure 2 Polymerisation of moisture curable terpolymers

The polymerisation is very straightforward and with high conversion of the silane. It results in a polymer with superior storage stability, well distributed silane groups, defined viscosity and which is free from any unreacted silane. In order to produce more flexible materials a third monomer, normally an acrylate, is introduced into the reactor, figure 2. In this way it is possible to produce curable "terpolymers" covering a flexibility range of 250-30 MPa. These polymers have an ability to accept high filler loadings.

1.2 Optimisation of processing characteristics

Crosslinking reactions occurring during the extrusion process will lead to an increased viscosity and might result in the formation of precured particles (scorch). These particles are unmeltable and can be seen as a contaminant reducing electrical and mechanical properties as well as negatively affecting surface finish and dimension stability. When using reactor copolymers it is possible to produce insulation having superior storage stability and product consistency. Compared with grafted materials, however, it was noticed at an early stage that the scorch sensitivity was worse. It was recognised that a rather high amount of unreacted silane monomer, 0.2-0.5%, was present in grafted polymers. This gave odour and health problems, but had one advantage. It improved the compound's scorch sensitivity. It is impossible to avoid crosslinking reactions taking place during extrusion, as small amounts of water will always be dissolved in the silane copolymer (10-30ppm) and more so in added masterbatches (300-2000ppm). It was proven that the unreacted monomer protected the polymer from precuring by

reacting with the water quicker than the polymer itself.

In fact unreacted monomer was added to the silane copolymer between 1986-88. It did indeed improve the scorch properties. On the other hand it created other problems due to its volatility, such as bad odour and the formation of glass like particles. It was also very difficult to maintain the correct level in the polymer due to rapid evaporation.

All these problems were solved 1989 by the introduction of a patented non-volatile scorch retardant additive⁽³⁾. The additive stopped more or less all molecular enlargements occurring during extrusion, again by reacting with water quicker than the polymer. The additive is non-volatile and is therefore present independent of storage time and storage conditions. The scorch retardant modified EVS copolymers based on this invention have been given the name Visico™.

2.3 Development of ambient curing and more environmentally sound catalysts

An additive reacting with virtually all the water present during extrusion made it possible to develop compounds with higher crosslinking speeds. An optimisation of the tin-based system in 1993 resulted in the introduction of LE4438, a catalyst masterbatch giving similar crosslinkability as the grafted systems but with superior processability due to the presence of the scorch retardant additive, figure 3. It was however, not possible to develop an ambient curing system based on tin catalysts.

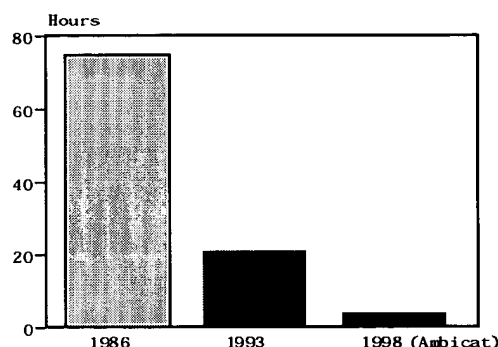


Figure 3 The development of the curing rate of Visico since its introduction in 1986. Time (hours) to reach 60% hot set elongation. 1.8 mm tape cured at 60°C in water.

Organo-tin compounds like DBTL have one further disadvantage. They are regarded as hazardous substances, as they inhibit the fundamental energy conservation process in mitochondria. Accordingly they are blacklisted by authorities and cable makers. So in addition to ambient curing, reducing

the impact on the environment and human exposure by changing to less harmful alternatives became the targets at the beginning of the 1990s.

A large number of possible candidates, such as non-tin based metal organic compounds, Lewis acids, acids, anhydrides and bases were screened. As can be seen in table 1, besides organo-tin substances, Lewis acids and organic acids showed interesting activity. The most promising substances were non-volatile and compatible sulphonic acids (SA). A class of substances now patented for ambient curing of silane crosslinkable polymers⁽⁴⁾.

	Number of substances tested	Ambient crosslinking 5 days, 50% rel. humidity %
DBTL	1	55
Lewis acids	11	0-63
Tin org. substances	31	0-62
Other metal organic substances	4	-
Organic substances Anhydrides	4	0
Acids	6	0-70
Bases	3	0
Others	6	-

Table 1 Achieved gel contents, measured by decaline extraction, for different type of crosslinking catalysts. (1.4mm tapes, 0.04mmol catalyst/kg)

A family of catalyst masterbatches based on this technology was introduced 1999. These masterbatches have indeed been found to crosslink low voltage cables at realistic speeds in ambient conditions. Accordingly they have been given the name Ambicat™. Some examples of its crosslinking ability are shown in figure 4. In ambient conditions, insulations below 1 mm are crosslinked within 48 hours at 50% relative humidity. The curing speed is primarily dependent of insulation thickness and temperature. The relative humidity plays also a role.

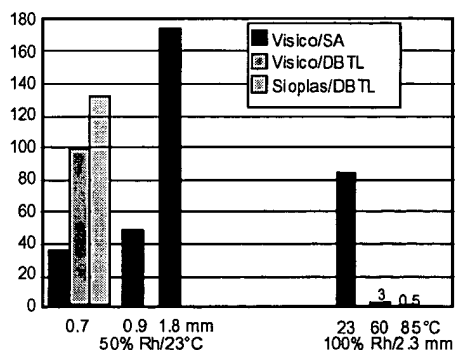


Figure 4 Influence of thickness, temperature, process and catalyst type on the curing speed. Time to reach 100% hot-set elongations for an insulated 1.5 mm² conductor. Visico = LE4423.

In the Ambicat family it has been possible to combine the fast curing speed with outstanding processability, scorch resistance, surface finish and little or no die drool figure 5. The absence of molecular enlargements during processing results in improved cable quality reduced scrap and a possibility to make short stops for tool changes etc., without any need for purging the material. Production campaigns of several weeks without cleaning are a reality. Absence of scorch and stable melt viscosity make it possible to produce very thin insulations, e.g. 0.2 mm wires, at line speeds over 1500 m/min. Further advantages are that no drying of the catalyst masterbatches is needed. In fact drying has a negative effect, as it consumes the drying agent present in this family of masterbatches.

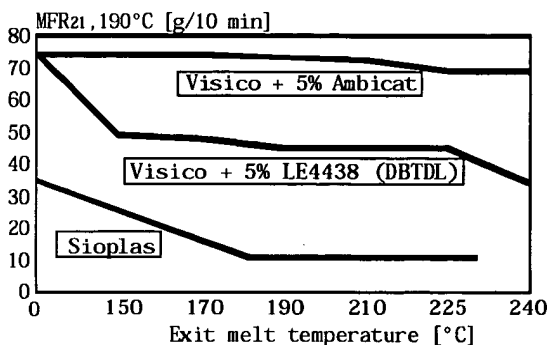


Figure 5 Influence of the scorch retardant additive as well as the catalyst type on melt viscosity changes (molecular enlargements) occurring during extrusion of moisture curable polymers

Three years of practical experience have shown that Visico/Ambicat offers significant production streamlining and reduced investments.

As the new catalyst is of acidic type, some important precautions are necessary. Metal soaps and basic components (high pH) such as stearates, and many fillers and pigments will deactivate the catalyst. To prevent catalyst deactivation during colouring, specially designed colour masterbatches have been developed in cooperation with the larger colour masterbatch suppliers.

In the original Ambicat masterbatches, problems with exudation of a powder, which under some circumstances could result in deposits on the feeder screw, were noticed. When storing cable drums for ambient curing in enclosed spaces a specific odour was also noticed. Both these problems have been found to be due to a reaction of some of the antioxidants present in the masterbatch with the sulphonic acid. A slight

modification of the stabilisation system has recently solved both these problems

2. LOW VOLTAGE APPLICATIONS

The main application of moisture curable polyethylene is as insulation for 1kV underground, industrial and overhead cables. Due to better electrical properties and a higher temperature rating these cables have a much higher power transmission capacity for a given conductor dimension compared with the traditionally used PVC cables. The improved electrical property results in significant material savings due to decreased insulation thickness. Besides lower cost, higher transmission reliability is reached, due to an increased overloading and short circuit resistance.

The disadvantages are that the production process is more complicated and expensive. Scorch retardant copolymers have simplified the extrusion process and made it possible to use ordinary PE/PVC extruders. Sauna or waterbath is, however, a heavy investment. The later curing step results in more complicated logistics, increased inventories and manpower costs.

2.1 Underground, building and industrial cables

By the introduction of the ambient curing technology most of these production complications have disappeared. Three years of practical experience have shown that low voltage cables, i.e. cables having an insulation thickness below 2,5mm, are crosslinked within realistic times at ambient conditions also during the dryer winter conditions in the northern part of the world. For many constructions it is also possible to run insulation and jacket in cascade.

For natural or coloured cables the above is reached by combining, LE4423 (Visico) with 5% of LE4476 (Ambicat). This fulfils the most common ageing requirements of 7 days, 135°C, on both aluminium and copper conductors. For the more stringent ageing requirements, 10 days, 150°C, an addition of 9% LE4476 is recommended. The ageing requirements are in more detail outlined in IEC60502-1. A terpolymer based zero-halogen flameretardant version is recently developed, FR4831/LE4435.

2.4 Overhead cables

For overhead lines the insulation is protected against sunlight by addition of carbon black to the insulation. In specifications such as HD626S1, levels over 2% are required. For such applications, an addition of 7% of the carbon black containing Ambicat masterbatch LE4472 is recommended. Such an addition will result in a carbon black content of 2.5%. In 24D or larger extruders

satisfactory dispersion of the carbon black is normally achieved.

For the more stringent UV requirements such as NF C33209 a larger amount (>4%) of extremely well dispersed carbon black is needed. Use of a carbon black containing Visico (LE4427) combined with 5% LE4476 is recommended.

3 MEDIUM VOLTAGE

3.1 Underground cables

In extending moisture cure technology into this voltage range it is particularly important to address the issues of cleanliness, smoothness, consistency of mechanical attributes and robust processing. MV moisture cured cables based on the Monosil and Sioplas process have been used since the late seventies. Despite this long track record, its use today is limited. This segment is still dominated by peroxide crosslinkable solutions with a market share of over 95%.

When using chemically grafted solutions, Monosil and Sioplas, considerable skill is required in order to maintain consistent mechanical and electrical performance, as it is necessary to maintain precise control of the chemical grafting reaction within either the compounding or cable extruder. Perturbations in the extrusion conditions, incoming raw materials or the addition of multiple components can result in significant variations in the final product. A particular concern when using the Monosil process is that the very nature of the manufacturing routes (chemical grafting of silane onto polyethylene within the cable extruder) makes it very difficult to assess the cleanliness of the crosslinkable insulation material prior to application to the cable. Sioplas technology enables quality control to be performed but exposes the materials to scorch from ambient moisture and limited storage stability.

The production of moisture curable semiconductives is more complicated than producing insulation in a grafting process. The semiconductive layer contains large amount of carbon black, which results in absorption of large amounts of water. This further reduce the storage stability of Sioplas based products even more. The high temperatures needed for the grafting operation induce scorch formation which significantly curtails production.

It is clear that the drawbacks faced by the Sioplas and Monosil technologies are significantly reduced when using Visico. The production of the insulation is accomplished within a purpose built chemical reactor, which is similar in design to those used for HV & EHV materials. On exiting from the reactor

the materials have the benefit of the quality control procedures that have been proven over many years for peroxide crosslinkable grades. Thus it is possible to assure cleanliness, reactivity and processability. The separate addition of catalysts, antioxidants and scorch retardant additives in a single polymer component greatly enhances the simplicity and reliability of the cable manufacturing process.

The introduction of a moisture curable "terpolymer" based bounded semiconductive in the mid 1990s was a further important step in making moisture curable copolymers a competitive MV solution.

In an ordinary silane copolymer the crystallinity is too high to incorporate the relatively high amount of carbon black needed in order to achieve semiconductive properties. Therefore a further comonomer which disturbs the ability of the molecule to crystallise has to be introduced. With peroxide crosslinkable semiconductives copolymers between ethylene and acrylates like ethyl, butyl or methyl acrylate, are normally used. To get a polymer base for a moisture curable screen a vinylsilane is added to such a polymerisation process, figure 2 The addition of a suitable carbon black gives a moisture curable semicon (LE0540), with excellent processability. LE0540 is intended as an inner screen and outer screen for bounded cable construction. It is not necessary to add any catalyst masterbatch to the semiconductors. The catalyst added to the insulation layer will quickly migrate to these layers.

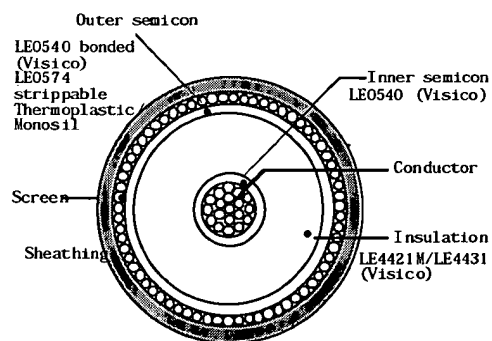


Figure 6 Copolymer based medium voltage solutions

When choosing a material class for use within a MV cable design it is important to consider the electrical performance. One convenient way of doing this is to consider the strength in a voltage ramp test. Figure 7 shows a comparison of the means (plus 90% confidence levels) for the virgin breakdown strength of Visico compared with a range of commercially used insulations based on peroxide and Monosil curing technologies⁽⁵⁾. These data

show higher (+6%) performance for Visico and reduced scatter – higher consistency.

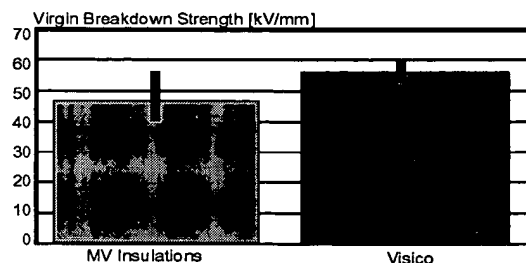


Figure 7 Virgin breakdown strength for Visico in comparison with cables with a range of insulation types, based of peroxide and Monosil technologies (mean and 90% confidence levels UNPEDE protocol)

Perhaps of more interest to the utility engineer is the life that may be expected. This attribute of a cable can most easily be examined by investigating the time to failure of equivalent designs under identical test conditions. Table 2 shows the result for such a test for cables manufactured with Visico and their Monosil analogue. In this case the Visico cable has at least 4 times the endurance (a longer time to the first failure) than the Monosil solution.

Cable	Time to first failure (weeks)
MONOSIL	4
VISICO	19

Table 2 Comparative wet ageing test of 24kV Cables. Water in conductor (90°C) and outside (55°C): Electric stress 9.2kV/mm.

These analyses clearly show the excellent electrical performance of Visico. Therefore the proven electrical, processing and consistency of Visico makes this technology extremely attractive for the production of consistent, reliable and cost effective MV power cables. As a consequence the volume of Visico materials used in MV applications choose a 2 - 4 fold annual increase. Visico is fast becoming the silane insulation choice.

3.2 Overhead cables

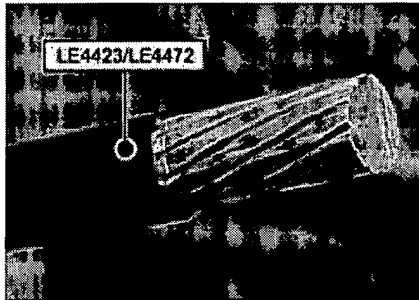
The second largest application for moisture curable copolymers is as covering/insulation of overhead conductors, a quickly growing segment.

3.2.1 Covered conductors

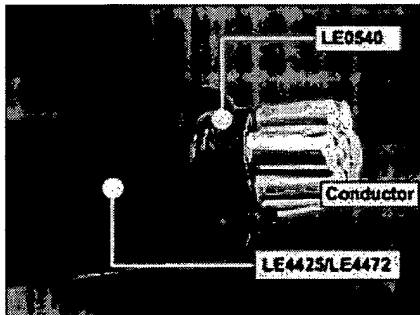
In northern Europe conductors rated up to 24kV has been used for since the late seventies. These cables are mostly utilised in the countryside and the

construction is meant to protect the cables against lightning, heavy winds, snowfall, touching trees etc. They are generally referred to as "covered conductors" or "free wires" and are not considered as insulated products. The reasons for switching to covered conductors are increased reliability and space reduction, as the distance between the conductors can be reduced by one third. The use of covered conductors has reduced the faults from 4.5 faults/100km for bare wires to 0.9 faults for covered conductors⁽⁶⁾.

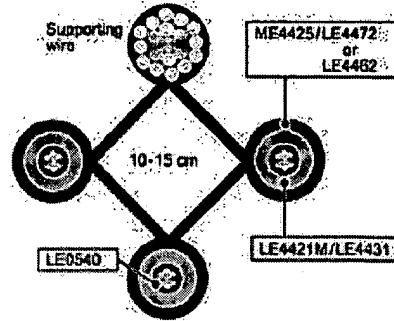
A covered conductor consists normally of a 3-4 mm moisture cured insulation containing 2.5% carbon black as UV protection. These cables were one of the first applications for the black ambient curing Masterbatch, LE4472 in combination with LE4423.



Covered conductor



Partial insulated cable



Spacer aerial cable

Figure 8 Moisture cured overhead cables

3.2.2 Partially insulated cables (PIC)

These cables are utilised in populated areas especially in tropical countries. The distance between the phases is approximately 30 cm. It's construction is outlined in figure 7. The insulation thickness varies between 2.8-5.4 mm. Due to the short distance between the phases the track resistant properties of these cables are defined. Measured in accordance with ASTM D 2303 the track resistance should be larger than >2.0kV, see table 3, a demand fulfilled with an ordinary copolymer such as LE4423 combined with LE4472 and utilising LE0540 as a moisture curable insulation screen. Peroxide crosslinkable solutions are also available using LE4217 in combination with the screens LE0592 or LE0595.

3.2.3 Spacer aerial cables (SAC)

SAC cables are used in the fast growing cities of the Far East, South America, Australia and the rural areas of northern Europe. The construction uses a frame where the three phases are fixed to each other by a rectangular spacer, see figure 8. The distances between the phases varies normally between 10-15cm. The phases consist of a conductor and a normally triple extruded layer consisting of an inner semiconductive layer, insulation and an outer carbon black filled weather and track resistant layer. The track resistant properties differ dependent on the country in which the technologies are used. Crosslinking, surface hardness, carbon black dispersion and decreased content improve the tracking properties, figure 9. Poor carbon black dispersion or a large amount of gels or foreign particles can create a serious discharge potential.

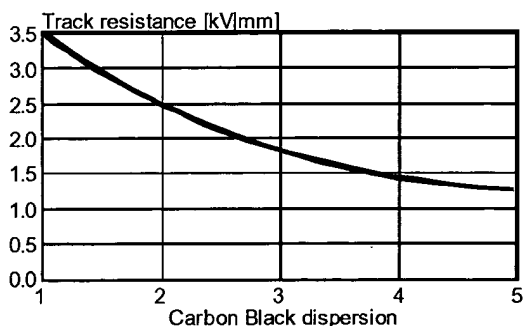
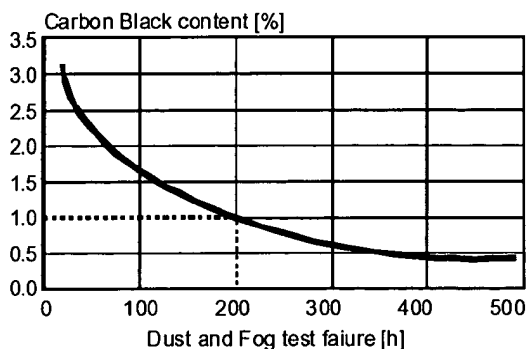


Figure 9. Influence of carbon black content and dispersion on track resistance (ASTM D 2332 and ASTM 2303)). Carbon black dispersion index measured in accordance with a slightly modified British Standard 2782 part 8.

As can be seen from table 3, LE4217 has similar tracking properties, 3.5kV, as the medium density based moisture cured material ME4425/LE4472. The positive effect of the harder material can be seen as it reaches the same tracking in accordance with ASTM D2303 at a less favourable carbon black content. LE4217 performs however better in the dust and fog test. ASTM 2332 compared with the moisture cured low density version, LE4423, the tracking is increased by 0.7kV to 3.5 kV and the resistance in the dust and fog test is increased from >200 to > 250 hours.

Properties	Standards				
	Covered conductors	PIC	LE4217	LE4423/LE4472	ME4425/LE4472
Polymer	Crosslinkable LDPE	Peroxide Crosslinkable LDPE	Peroxide Crosslinkable LDPE	Visco™	Visco™
Carbon black content	>2.5%	>0.5%	0.5%	2.5%	2.5%
Track Resistant ASTM D2303	No	>2.0 kV	3.50 kV	2.80 kV	3.50 kV
Track Resistant ASTM D2132	No	>200 h	>300 h	>200 h	>250 h
AC Breakdown	>5 kV/mm	>5 kV/mm	>22 kV/mm	>22 kV/mm	>22 kV/mm
Heat Elongation	>175%	>175%	60.80%	60%	40%
Tensile Strength	>12.5 MPa	>12.5 MPa	22 MPa	17 MPa	25 MPa
Elongation at break	>250%	>250%	500%	350%	380%
Variation of mechanical properties after 7 days at 135°C	<25 %	<25 %	<25 %	<25 %	<25 %
Variation of mechanical properties after 2000h UV ageing	<10 %	<10 %	<10 %	<10 %	<10 %
ESCR	no cracks	no cracks	>2000 h	>1000 h	>1000 h
ASTM D 163	1000 h	1000 h			

Table 3 Track resistant performance for different curable compounds

The most severe tracking demands are in the Brazilian specification (ABRADEE CODE – 3.2.18.23.1). A modified version of the ASTM D2303 is used. In order to stress the tracking, the surface shine of the cable is destroyed with 600-grit sandpaper. The cable must then withstand 2.75 kV without tracking. ME4425 has to be combined with a specially developed catalyst masterbatch, LE4462, in order to pass this requirement. This masterbatch consists of a tin catalyst in combination with an UV stabiliser and a smaller amount of carbon black.

4 FUTURE OUTLOOK

4.1 Low Voltage

A common problem when extruding moisture curable compounds onto cold conductors utilising pressure or semi-pressure dies are reduced mechanical properties. The reason is that a heavily oriented molecular layer is formed close to the conductor. Even though preheating the conductor or changing to tube on die solves the problem, it creates complications. Ongoing work is trying to optimise the copolymer characteristics to make them less sensitive to orientation/freezing.

Terpolymers based on ethylene, acrylate and vinyl silane are not only an interesting polymer base for filled moisture curable compounds like semiconductives and halogen free flame retardant compounds. They can also be used for the production of flexible cables.

4.2 Medium Voltage

Work in this area has shown that acidic and tin based catalyst systems give similar electrical performance, in both the short and long term tests. In the coming years a transfer from tin catalysts to

the more productive acidic type of catalyst is therefore expected.

The curing speed seems, however, not to be so dramatically affected as for low voltage applications. For the thicker medium voltage cables a big difference in the curing of the surface layer is seen, see figure 10. For the layer close to the conductor a similar curing speed is observed.

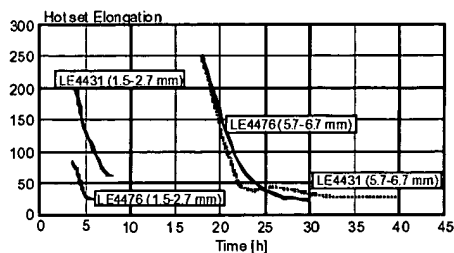


Figure 10 Comparison of crosslinking speed at different distances from the insulation surface between a tin based catalyst masterbatch (LE4431) and a SA based masterbatch (LE4476). Crosslinking performed in water at 90°C

A way to overcome this weakness is to use a less crystalline and a more polar polymeric system, e.g. a terpolymer. In Table 4 the curing speed of the inner layer of a terpolymer consisting of 17% butyl acrylate, LE4424, is compared with the traditional EVS copolymer, LE4421M. Clearly such a solution will reduce the curing time in a waterbath by about four times.

Hot set elongation on a 1,2 mm micron cut between 2,9- 4-1 mm from the surface

Water bath	2h 90°C (%)	4h 90°C (%)	8h 90°C (%)	12h 90°C (%)	14h 90°C (%)
LE4421M					
+5% LE4476	Fail	Fail	Fail	45	35
LE4424 (17%BA)					
+5% LE4476	Fail	20	15	15	15
LE4424 (17%BA)					
+5% LE4431	Fail	Fail	-	-	Fail
Ambient (23°C, 50% humidity)	5 weeks %	6 weeks %	12 weeks %	17 weeks %	
LE4421M					
+5% LE4476	Fail	Fail	Fail	215	
LE4424					
+5% LE4476	65	50	-	-	

Table 4 Comparison of crosslinking speed between a copolymer and a terpolymer (17%BA) of a medium voltage insulation. Hot set elongation of microtome cuts between 2.9-4.1 mm from the surface of the insulation.

Tin based 10 kV cables are cured for about 24-48 hours in a waterbath at 70-80°C. Utilising Ambicat and terpolymer technology this will be reduced to 6-9 hours, similar to that used for tin based low voltage cables. Terpolymer based cables will be

more flexible than the copolymer based cables. The 17% BA terpolymer used in this example have a flex modulus of 40 MPa. This can be compared with LE4421M, which has a flex modulus of 200 MPa.

A further possibility is to develop strippable outer semiconductive layers based on the high acrylate containing terpolymers.

5. CONCLUSIONS

Fifteen years development of moisture curing copolymer technology has resulted in a scorch retardant additive, which reacts with the water present during the extrusion operation. Besides improved processability, scorch resistance and die drool performance it has resulted in the possibility to develop more environmentally safe and ambient curing catalysts for low voltage applications.

Three years of practical experience have shown that the combination of a scorch retardant copolymer (Visico) with an ambient curing masterbatch (Ambicat) have indeed show that curing takes place "automatically", i.e. without any need for sauna or waterbath. Resulting in simplified production logistics, reduced inventories as well as decreased manpower and investment costs.

Terpolymers consisting of ethylene, vinylsilane and acrylate are today the polymer base for moisture curable semicons and halogen free compounds. In the future this technology might be utilised for production of flexible cables. Combined with an acidic based catalyst they show interesting properties with respect to crosslinking thicker objects e.g. medium voltage cables, 3-4 times faster than the solutions which exist today.

Fifteen years of focused development have given Visico/Ambicat a market position as the most versatile and cost effective moisture curing solution.

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